

## A Disposable Single-Use Electrochemical Sensor for Detection of Resorcinol Based on Electrochemically Activated Screen Printed Carbon Electrode in Hair Dyes

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A simple and effective amperometric sensor based on activated screen printed carbon electrode (ASPCE) was successfully fabricated for the electrochemical determination of resorcinol (RC). The ASPCE was fabricated by electrochemical method and it was characterized by using various numerous technique. The electrocatalytic behaviour of the fabricated ASPCE were examined by the cyclic voltammetry (CV) and amperometric method (I-T). Remarkably, ASPCE exhibited an excellent electrocatalytic activity in terms of decrease the potential and increase the anodic peak current for the detection of RC when compared to bare SPCE. The ASPCE was successfully determined the GA and it showed linear response range from 1 to 49.67  $\mu\text{M}$  and the low detection of limit (LOD) of 0.288  $\mu\text{M}$ . Furthermore, the proposed sensor electrode was revealed an excellent selectivity and good stability. To the best of our knowledge, this fabricated electrode is cost effective and good electrochemical performance towards the determination of RC when compared with previously reported other chemically modified electrodes.

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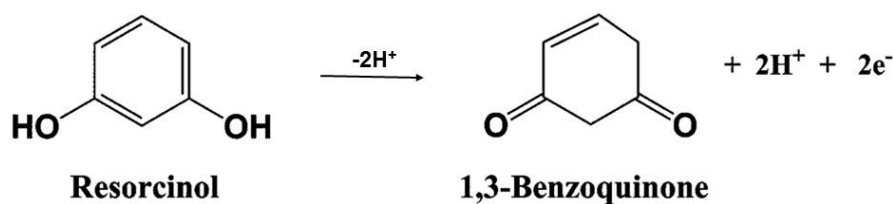
**Keywords:** Resorcinol, Cyclic voltammetry, Electrochemistry, Activated screen printed carbon electrode.

## 1. INTRODUCTION

Nowadays, environment pollution become a serious and crucial problem in worldwide to the human body even at low concentrations [1]. Phenolic compounds, as a class of organic compounds, have a similar functional groups which are commonly resistant to biological degradation [2]. These compounds are continually used as important chemicals in different manufacturing industries such as cosmetics pesticides, gas works, tanning, medicine, paper mills pharmaceutical and photography applications [3,4]. Resorcinol (RC) is a one type of phenolic compounds with hazardous nature. RC is used in the manufacture of cosmetic products such as hair dye, skin peels, bleaches [5]. It is easily penetrating through the skin, which can cause cyanopathy, catarrh dermatitis, scalp, convulsion, kidney damage, liver infections and even death [6]. In addition, accident intake of RC directly affects the respiratory failure, cyanosis, central nervous system, including unconsciousness, drowsiness and seizures [7-10].

Until now, a numerous of techniques have been developed for the determination of RC including high performance liquid chromatography, spectrophotometric, microchip capillary electrophoresis, quartz crystal microbalance, chemiluminescence, surface plasmon response, spectrofluorometric and florescence [10]. Among all, electrochemical techniques provide many advantages such as high sensitivity, selectivity, time saving, moderate cost, simplicity and portability [11-13].

There are different types of electrodes have been developed and applied for the electrochemical detection, particularly carbon based materials, such as carbon ionic liquid electrodes [14], graphite electrodes [15], carbon paste electrodes [16], glassy carbon electrodes (GCE) [17], carbon fiber microelectrodes [18], and screen-printed electrodes [19]. Among these, Screen-printing electrode (SPCE) a well-designed electrode fabricated technique with flexibility, highly reproducible manner, easily disposable and mass production, [20]. SPCE has revolutionized not only by cost effective but also good electrocatalytic properties [21]. Compton's group reported that the edges-plane pyrolytic graphite is more active than the original defect/edge plane sites. For more versatile applications, the surface of electrode was chemically or electrochemical modified with functional active compounds. The surface of electrode was functionalities with oxygen groups and generation of edge plane likes sites [22-24]. The work deals with fabrication of electrode with economic view and selective determination of RC. Hence, in the present study, for the first time we reported the preanodized SPCE (ASPCE) for the electrochemical detection of RC. The electrochemical behaviour of ASPCE was compared with previously reported modified electrodes in table 1. The ASPCE was characterized by different physicochemical characterizations.



**Scheme 1.** Oxidation mechanism of Resorcinol (RC)

## 2. MATERIALS AND METHODS

### 2.1. Materials and instruments

Screen printed carbon electrode (working area = 5 mm) were purchased from Zensor R&D Co., Ltd, Taiwan. Resorcinol (RC), ascorbic acid (AA), uric acid (UA), gallic acid (GA) acetaminophen (AC), hydroquinone (HQ), insulin, ferulic acid (FA), catechol (CT), morin (MR), salicylic acid (SA), sodium chloride ( $\text{Na}^+$ ), potassium chloride ( $\text{K}^+$ ), zinc chloride ( $\text{Zn}^{2+}$ ), copper chloride ( $\text{Cu}^{2+}$ ), calcium chloride (Ca), magnesium chloride ( $\text{Mg}^{2+}$ ), sodium hydrogen phosphate ( $\text{NaHPO}_4$ ), sodium hydroxide (NaOH), sodium dihydrogen phosphate ( $\text{NaH}_2\text{PO}_4$ ) and all other chemicals were obtained from Sigma-Aldrich and used without any additional purification. Double distilled (DD) water were obtained from a Millipore purification system.

The structure was examined by using scanning electron microscopy (SEM Hitachi S-3000 H). Raman spectra were recorded using a Raman spectrometer (Dong Woo 500i, Korea) equipped with a charge-coupled detector. Electrochemical impedance spectroscopy (EIS) by ZAHNER Scientific Instruments (THALES software package). Cyclic voltammetry (CV) and amperometry experiments were executed by using CHI 750A work station (*Inc. Made in U.S.A.*). Electrochemical techniques were studied in a conventional three electrode system using ASPCE as a working electrode (area 5 mm), a platinum (Pt) wire as a counter electrode and saturated Ag/AgCl (saturated KCl) as a reference electrode. All the electrochemical measurements were carried out at room temperature.

### 2.2. Methods

The ASPCE was fabricated by using our previously reported method with minor modification [25]. In briefly, 0.1 M of KOH was prepared by using double distilled (DD) water. Then, the untreated- SPCE was immersed into an electrochemical cell containing 0.1 M KOH, followed by the constant potential range from 0 to 2.0 V up to 12 cycles for anodizing SPCE. Pretreated SPCE (ASPCE) was dipped into water dried in oven for hour. Finally, the obtained ASPCE was used for further electrochemical measurements.

## 3. RESULTS AND DISCUSSION

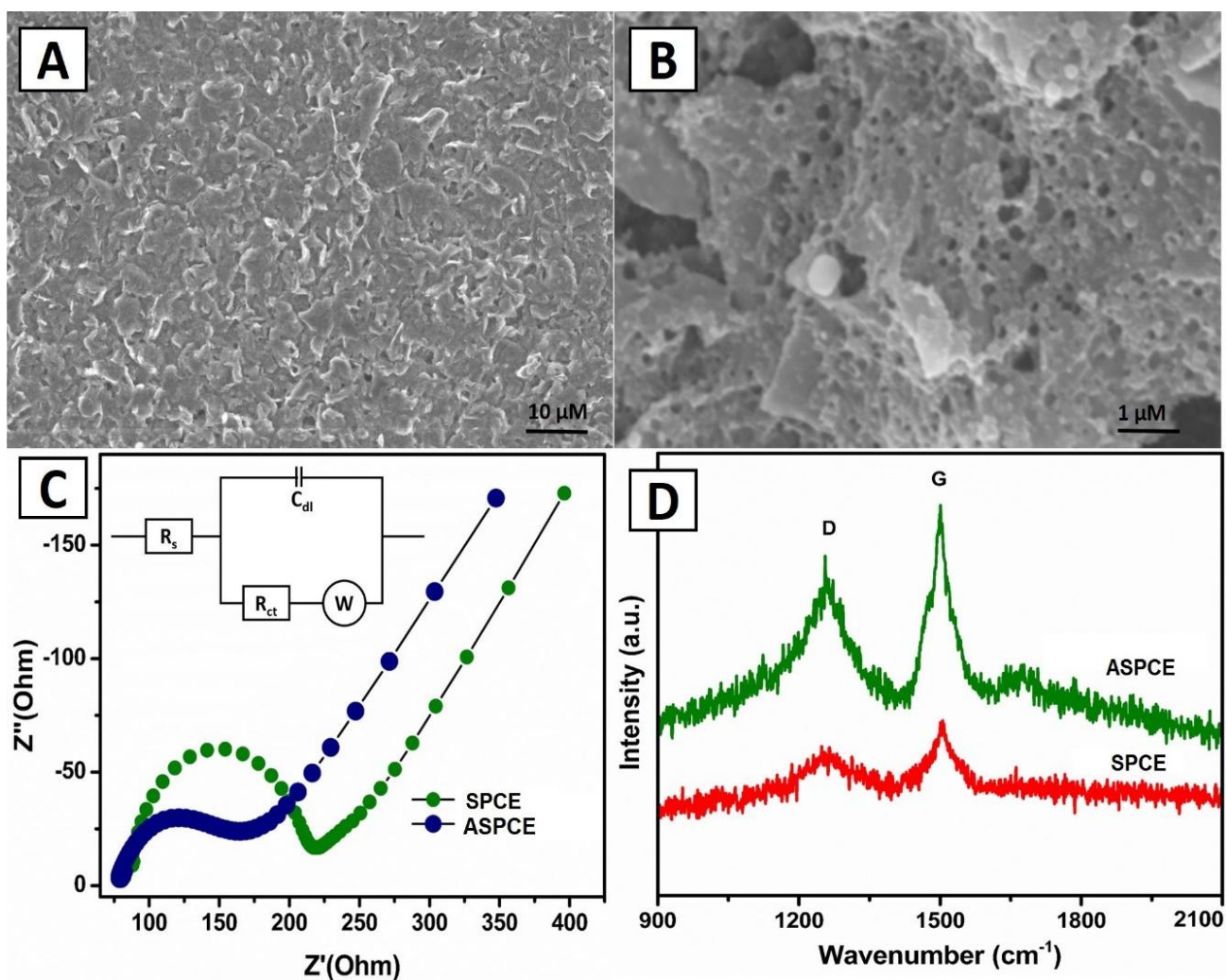
### 3.1 Characterization of ASPCE

Fig. 1 A & B shows the SEM images of SPCE and ASPCE. From the Fig. 1A, it was seen that that the before anodizing SPCE shows rough and aggregated flake like surfaces [19]. After anodized the SPCE morphology of the electrode was transferred into honey comb like structure, which reals that the SPCE was successfully anodized (ASPCE).

Electrochemical impedance spectroscopy (EIS) is an effective analytical technique to estimate the electron charge transfer process, which is mainly depend upon the solution resistance ( $R_s$ ) and charge transfer resistance ( $R_{ct}$ ) between electrode/electrolyte interfaces. Here,  $R_{ct}$  value is measured at

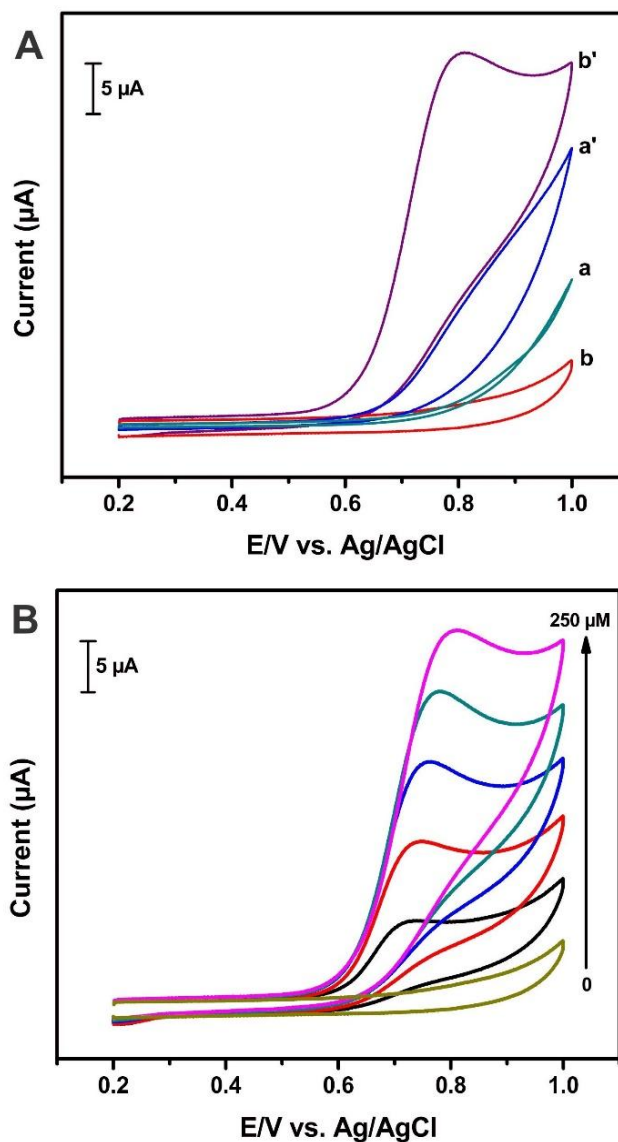
lower frequency region and  $R_s$  is measured at higher frequency region of semicircle. In general, the radius of semicircle is directly proportional to  $R_{ct}$ . Fig.1C shows the Nyquist plots of the (a) SPCE and (b) ASPCE in the supporting electrolyte of 0.1 M KCl solution containing 5 mM of  $[\text{Fe}(\text{CN})_6]^{-3/4}$  with applied frequency range from 100 kHz to 100 Hz and amplitude of 10 mV. In result of this experiment, ASPCE exhibit  $R_{ct}$  values of about 78.6  $\Omega$  which is comparatively lower than SPCE (138.3  $\Omega$ ). The corresponding electrical equivalent Randles circuit is shown in inset of Fig.1C which consists of charge transfer resistance ( $R_{ct}$ ), solution resistance ( $R_s$ ), double layer capacity ( $C_{dl}$ ) and Warburg impedance ( $W$ ).

Fig. 1D displays the Raman spectra for SPCE and ASPCE with the appearance of the two major peaks. The D band is reveals to the A1g breathing mode of the disordered graphite structure and G band is exposes the doubly degenerate graphite [26]. Furthermore, Intensities ratio ( $I_D/I_G$ ) of SPCE was changed 1.169 to 0.89 after anodizing SPCE (ASPCE) which is directly proportional to the disorder in its hexagonal lattice. This intensity changes confirms the ASPCE have good conductivity.



**Figure 1.** SEM images of (A) SPCE (B) ASPCE (C) EIS of SPCE and ASPCE (D) Raman spectra of SPCE and ASPCE

## 3.2 Electrochemical behavior of RC



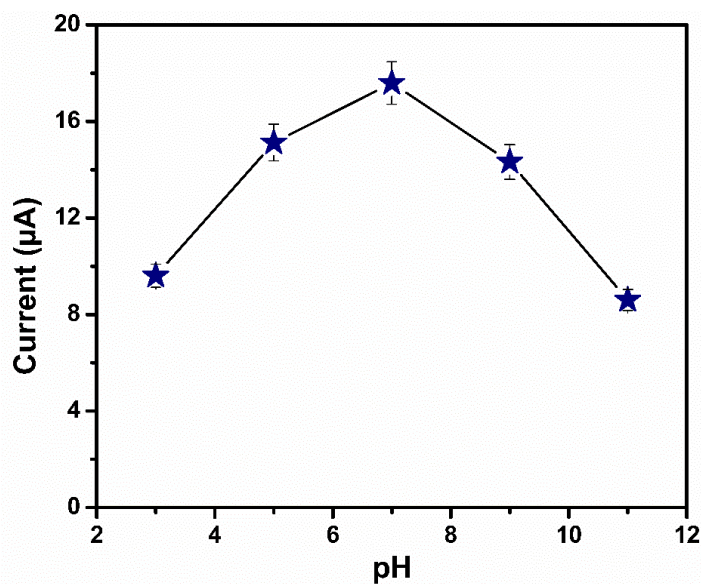
**Figure 2.** (A) CV response of (a) SPCE (b) ASPCE in the absence and (a') SPCE (b') ASPCE in the presence of 250  $\mu\text{M}$  of RC containing 0.05M PBS (pH 7.0) at a scan rate of 50 mV/s. (B) CV response of RC on ASPCE in various addition ranges from 0 to 250  $\mu\text{M}$  containing pH 7.0 (0.05M PB solution) at a scan rate of 50 mV/s.

The electrochemical behavior of modified electrodes was investigated by using CV. Fig. 2A shows the electrochemical performance of RC on (a) SPCE (b) ASPCE in the absence and (a') SPCE (b') ASPCE in the presence of 250  $\mu\text{M}$  RC containing 0.05M pH 7.0 (PB solution) at a scan rate 50 mV/s. CV behaviour in the absence of RC at SPCE (curve a)/ASPCE (curve b) shown in Fig. 2A. It clearly shown that, there is no peaks was observed in the absence of RC On another hand, in the presence of 250  $\mu\text{M}$  RC at SPCE shows a weak anodic peak was obtained and the oxidation peak current is very lower when compared to ASPCE. On the other hand, in the presence of 250  $\mu\text{M}$  RC at ASPCE shows a well-shaped oxidation peak was observed at the potential of 0.76 V, which indicate

that the oxygen functionalities in edges plane enhance the peak current for the detection of RC. For comparison, the obtained anodic peak current at ASPCE is 6 folds higher when compared to SPCE. This results clearly shows that the ASPCE have excellent electrocatalytic activity towards RC than the SPCE. Fig. 2B displays the CV curve of various additions of RC (0-250 $\mu$ M) in N<sub>2</sub> saturated 0.05M pH 7.0 (PB solution) at a scan rate of 50 mV/s. The oxidation peaks current was increased with increasing the concentrations of RC, reveals that the good electroactive species on the ASPCE. This obtained results indicates the ASPCE could be superior electrochemical behaviour towards RC detection.

### 3.2.1 Influence of pH

In order to evaluate the effect of pH (3.0-11.0) was examined carefully in the presence of 250  $\mu$ M of RC at ASPCE by CV and the results are shown in Fig.3. The anodic peak current of RC was gradually decreased negative potential in the pH (pH 3.0 and 5.0) and the peak shifted towards more positive values when from increasing the pH value (pH 9.0 and 11.0). Notably, pH 7.0 have a superior electrocatalytic activity. Hence, the pH 7.0 was chosen for the further electrochemical determination of RC.

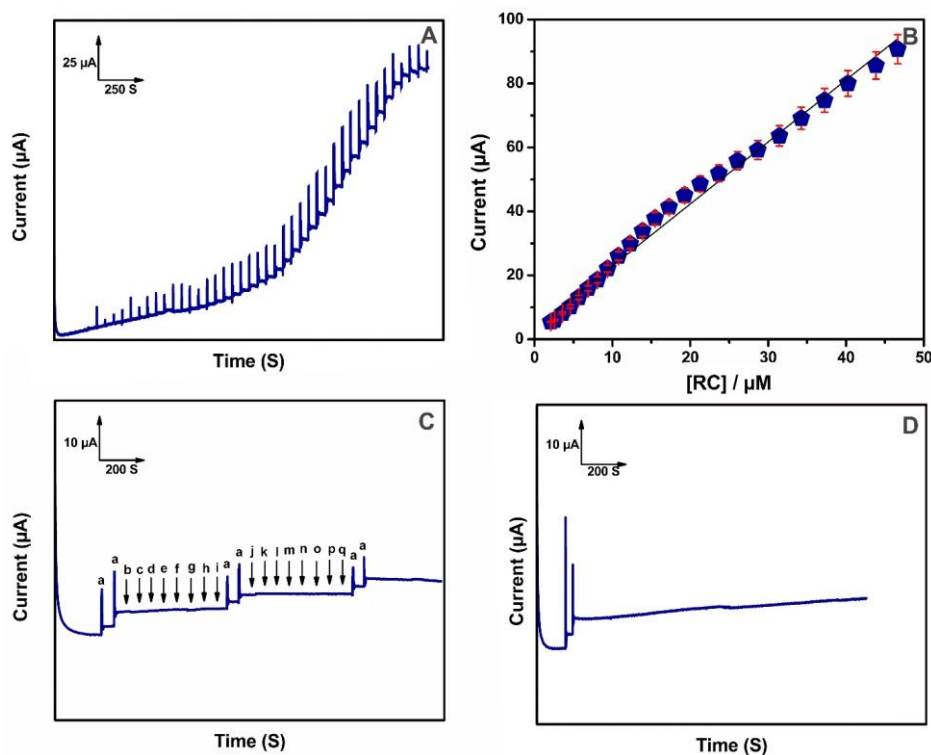


**Figure 3.** CV response of RC on ASPCE in various pH ranges from 3.0 to 11.0 (0.05 M PB solution) in the presence of 250  $\mu$ M of RC at a scan rate of 50 mV/s.

### 3.2.2 Amperometric detection of RC

Fig. 3A shows the amperometric I-T response of ASPCE for the successive additions of RC for various concentrations ranging from 0.06  $\mu$ M to 110  $\mu$ M for consecutive injected at regular intervals of time (50 s) into constantly stirred 0.05 M pH 7.0 (PB solution). The applied electrode potential was held at 0.76 V, with rotation speed was fixed at 1200 rpm. The anodic peak current of RC was increased with increasing the different concentrations. The anodic peak current was plotted against the

concentration of RC and shown in Fig. 3B, it exhibits the linearity from 1 to 49.67  $\mu\text{M}$  with the linear correlation coefficient of  $R^2 = 0.9964$ . From the plot, the lower detection limit (LOD) of RC was calculated to be 0.289  $\mu\text{M}$ . The obtained electro-analytical parameters at ASPCE such as linear ranges and (Limit of detection) LOD are summarized in Table 1 [3-44]. Furthermore, based on the obtained results we conclude that the ASPCE plays an important role for the oxidation of RC due to activation of SPCE as well as edge of oxygen functionalities in ASPCE [45]. The oxygen functionalities groups in ASPCE provides more active sites for the oxidation of RC and it enhanced the electrocatalytic activity of RC. ASPCE considerably reduces the peak potential of RC which leading to power consumption and also low cost electrode compared to other modified electrode materials like metal oxide, graphene oxide, ionic liquid, bio polymers, nano metals, MWCNT, SWCNT and graphene, conducting polymers [3-44]. The presented electro-analytical parameters at ASPCE towards RC which are very comparable with previously reported RC sensors.



**Figure 3.** (A) The amperometric response of ASPCE with different concentration of RC in pH 7 (0.05 M PB Solution) from 0.3  $\mu\text{M}$  to 113.8  $\mu\text{M}$ , (B) The calibration plots of peak current (RC) vs. its concentration, (C) The amperometric response of RC with the 10-fold excess interfering compounds such as RC (a), AA (b), UA (c), AC (d), CT (e), GA (f), FA (g), SA (h), MR (i), insulin (j), DA (k),  $\text{Zn}^{2+}$  (l),  $\text{Cu}^{2+}$  (m), Ca (n),  $\text{Mg}^{2+}$  (o),  $\text{K}^+$  (p),  $\text{Na}^+$  (q), (D) The amperometric response of RC in ASPCE attaining the steady state current.

### 3.2.3 Interference study

In order to investigate the selectivity of the proposed sensor, amperometric I-T technique was performed for the determination of RC in the presence of potentially interfering biological compounds

and common metals. Fig. 3D shows the amperometric I-T responses of RC with 10 excess fold concentration of interfering ions such as RC (a), AA (b), UA (c), AC (d), CT (e), GA (f), FA (g), SA (h), MR (i), insulin (j), DA (k),  $Zn^{2+}$  (l),  $Cu^{2+}$  (m), Ca (n),  $Mg^{2+}$  (o),  $K^+$ (p),  $Na^+$  (q). This observed results indicated their no such mentionable response for the interference compounds. This study confirmed that ASPCE was highly suitable for the detection of RC.

### 3.2.4 Stability

The stability of the ASPCE were examined by using amperometric I-T technique. Fig.3D displays the amperometric I-T responses of RC oxidation at ASPCE with the addition of 100  $\mu$ M of RC in 0.05 M pH 7.0 (PB solution) for the steady state current response up to 2300 s with a working potential of 0.76 V. The ASPCE shows 91% retention response from the initial current response. The obtained results reveals that the ASPCE have acceptable stability and it can be used for the real time monitoring.

**Table 1.** comparison on the analytical performance of ASPCE with previously reported detection of RC.

Electrode	Linear range ( $\mu$ M)	LOD ( $\mu$ M)	Ref
GR-CHI/GCE	1.3-133.4	0.350	3
PEDOT-Gr	1-250.0	0.160	27
MOF/CHI-ERGO/GCE	1-200.0	0.33	28
PANI/ $MnO_2$ /GCE	0.2-100.0	0.09	29
SWCNT/GCE	0.4-10.0	0.3	30
Au-PdNF/rGO/GCE	2-100.0	0.7	31
MCNTs/GCE	5-80.0	1.0	32
ZnO/graphene	0-700.0	1.0	33
Gr-Chitosan/GCE	1-550.0	0.75	34
ZnO/carbon cloth	2-385.0	7.2	35
Ag doped $TiO_2$	0-15.0	24.9	36
PAIUCPE	4-400.0	120	37
MWCNTs- $NTiO_2$	1.6-160.	32	38
MWCNT/b-CD/CILE	2.6 -900.0	0.9	39
Nitrogen-doped CNT/GCE	50-1000.0	5.64	40
SWCNH/GCE	1-100.0	0.5	41
GR-CN-Cu	0.1-320.0	0.13	42
MWNTs-IL/GCE	1.9-145.0	0.38	43
ZnO/GR/Ta	0-700.0	1.0	44
ASPCE	1 -49.67	0.289	This work

**Abbreviations:** GR-Graphite, CHI-Chitosan, Gr-Graphene, MWCNT-Multi walled carbon nanotube, SWCNT- Single walled carbon nanotube, IL-Ionic liquid, Ag-Silver, ZnO-Zinc Oxide,  $TiO_2$ - Titanium



dioxide, b-CD- Beta-cyclodextrin, RGO-Reduced graphene oxide, ERGO- Electrochemically reduced graphene oxide, MnO<sub>2</sub> - Manganese dioxide, PEDOT- Poly(3,4-ethylenedioxythiophene), Cu-Copper nanoparticles, MOF- Metal-Organic Framework Materials, CN-Nitrogen doped carbon, PAIUCPE- Pre-anodized inlaying ultrathin carbon paste electrode, CILE- Carbon Ionic Liquid Electrode, PdNF- Palladium nanofiber.

### 3.2.5 Real sample

To measure the real time application of fabricated sensor was measurement of RC in hair dyes by amperometry I-T technique. The experimental conditions are similar to Fig. 3A. The standard addition method was used for calculation of recovery of RC in hair dyes. A known concentration RC containing hair dye was injected into the supporting electrolyte solution and the obtained results are listed in Table 2. The proposed sensor showed the good recovery values ranges from 97.6 % and 106 % with acceptable relative standard deviations (RSD). Hence, the obtained RSD vales indicated that the proposed sensor exhibited significant recovery values in real samples.

**Table 2.** Detection of RC in hair dyes

Hair dyes	Added ( $\mu\text{M}$ )	Found ( $\mu\text{M}$ )	Recovery (%)	RSD (%)
Sample 1	20.0	21.2	106.0	2.9
	30.0	29.3	97.6	3.6
Sample 2	20.0	20.2	101.0	3.1
	30.0	30.1	100.3	3.8

## 4. CONCLUSION

In conclusion, we activated SPCE through KOH by effective and simple electrochemical method. ASPCE was successfully investigated by SEM, Raman Spectra, EIS and electrochemical techniques. It is one of best electrochemical platform for detection of RC. The fabricated ASPCE displayed the fast response, good sensitive, including low detection limit, stability and excellent selectivity in the presence of potential interfere metal ions and biological molecules. Additionally, the fabricated sensor was successfully used for the determination of RC in real samples and exhibited good performance. In future, ASPCE is used for furthermore application in various field.

### ACKNOWLEDGEMENTS

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